EVALUATION OF THE PHYSIC-CHEMICAL PROPERTIES OF AGRO-WASTES DERIVED ACTIVATED CHARCOAL AS A POTENTIAL FEED ADDITIVE IN POULTRY PRODUCTION.

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Abstract
The yearly production of agro-wastes from different agricultural operations in Nigeria is in abundance and poses major environmental concerns if not properly managed. Thus, such waste resources could be processed into sorbent products and characterized for appropriate applications. A blend of pig dung, palm kernel shell and bamboo chips were carbonized, and the resultant activated charcoal (AC) was evaluated for biophysical properties such as bulk density (BD), water holding capacity (WHC), specific gravity (SG), oil adsorption capacity, pH, ash, and carbon content. The AC was also assayed for its concentration of minerals. Results showed that AC had 0.871 g/cm^3 BD, 0.879 % WHC, 0.725 SG, and was mildly alkaline (8.49). Activated charcoal also yielded high percentage carbon content (75.35 %) and low ash content (13.13 %). The elemental composition of AC had higher P and K, Fe, and Mn compared to other macro and micro minerals respectively. The order of mineral abundance in the AC produced was P>K>Ca>Mg>Na> Fe> Mn> Zn> Cu, and Cl and could serve as a feed additive in livestock feeding.
Key Words: Activated charcoal, Agro-waste, Physic-chemical, Mineral concentration, Pig dung.

Introduction

The intensification of agricultural production in response to the growing world population often generate substantial quantities of agro- residues and wastes more than the primary products from such operations. Agricultural wastes are composed majorly of crop waste (stubbles, fruits, and vegetables etc.), animal waste (manure, waste feed and animal carcasses), and by-products of food processing with little or no economic value to the farmer (Okoli, 2020). Nigeria as a country also generates her fair share of these wastes from crop, and livestock production. With an aggregate crop production of 93.3 million tons of major cash crops, and an estimated 285.1 million tons of crop waste, and manure from livestock yearly, the country tends to generate more quantity of stubbles, straws, chaffs, husks, offal's, and animal manure than the actual food crops and animal products (Sillar, 2000; ECN, 2008). The quantity of these agricultural residues utilized either as feed for livestock, fertilizer for crop production or heat generation is however negligible with a greater percentage dumped at landfills and/or incinerated due to lack of investment in waste management technologies. The piles of pig dung for example has been a major source of litigation and confrontation within the communities where such farms are sighted due to the environmental pollution. Therefore, processing these waste resources into activated charcoal, and biochar products for agricultural, and industrial applications could be a worthy option (Yahya *et al.,* 2015).

Activated charcoal is a very porous, non-soluble adsorbent, produced from the incomplete combustion of agricultural residues, and waste under controlled oxygen environment. It is chiefly characterized with enormous absorptive, and bacteriostatic properties owing to its large internal surface area, which enables it to adsorb toxins, gases, anti-nutrients, and bacteria in contaminated livestock feeds (Kana *et al.*, 2011; Mgbeahuruike *et al.*, 2018), manure litter/soil (Maurer *et al.*, 2017; Borchard *et al.*, 2019), pollutants in industrial effluents, and in drinking water filtration (Edward *et al.*, 2010; Gwenzi *et al.*, 2017). Studies are increasingly reporting the performance benefits of activated charcoal (AC) supplementation in poultry (Prasai *et al.*, 2018; Kalus *et al.*, 2020), and pig diets (Chu *et al.*, 2013; Van Chao *et al.*, 2016). Ingested toxins effectively bind to activated charcoal, thus mitigating adverse reactions within the gut system (Anjaneyulu *et al.*, 1993), while promoting digestive efficiency, and growth in animals.

AC supplementation could also modify gut, and soil microbiota by causing reductions in the pathogenic load, and increased proliferation of beneficial microorganisms in such habitat (Joseph *et al.*, 2015; Rattanawut *et al.*, 2017; Willson *et al.*, 2019). The rich mineral elements contained in AC can electrically support microbial growth required in feed degrading, and biochemical reactions (Shi *et al.*, 2016), while meeting the mineral requirements for crop, and livestock production. Studies also showed that AC could enhance soil carbon sequestration, soil fertility, and soil pollution remediation (Xiao *et al.*, 2018; Dang *et al.*, 2020).

Several studies have demonstrated the utilization of agricultural residues, and wastes such as rice husk (Alvarez *et al.*, 2014), palm kernel shells (Evbuomwan *et al.*, 2013), corn cob (Kana *et al.*, 2011), coconut shells (Shaheed *et al*., 2015), bamboo (Chu *et al.*, 2013), and manure (Lima and Marshal 2007; Shakya and Agarwal, 2017) as promising raw materials for AC production. There are two methods of activated charcoal production from agro-residues, namely, physical, and chemical activation methods. However, the choice of material for AC production will depend on the availability, volatility, carbon content, ash yielding capacity, and the intended use of the AC to be produced (Tadda *et al.,* 2016). This is because, AC produced from various raw materials under similar conditions have varying physic-chemical properties which affects its adsorptive quality (Mdoe, 2014).

In this study, pig manure was blended with palm kernel shell (PKS), and bamboo to produce AC as a possible additive in livestock feed production. The controlled combustion of pig dung together with fire accelerants like PKS, and bamboo at appropriate blends have been shown to improve the combustion value of the dung during activated charcoal or briquette production (Iregbu, 2014). Thus, the purpose of this study is to produce, and characterize activated charcoal from the waste resources. The physic-chemical properties of the resultant AC product such as bulk density (BD), specific gravity (SG), water holding capacity (WHC), pH, fixed carbon, and ash content etc., as well as its potential mineral concentrations were assayed to determine its potentials, and possible utilization as a feed ingredient. This is because, the physic-chemical properties of feed ingredients influence feed acceptability, and consumption in animals (Makinde and Sonaiya, 2007), and therefore, should be considered when selecting ingredients for diet formulation.

Materials and Methods

Collection, Preparation and production of Activated Charcoal: Freshly voided pig dung was collected into a clean plastic container from a local piggery farm located at Olakwo Enyiogugu in Aboh Mbaise Local Government Area (LGA), Imo State. The dung was spread on a clean slab, and sun-dried. Palm kernel shells collected from a local oil mill also located in the same town were washed, sieved, and sun – dried. Bamboo wood from the same area was harvested, and cut into small chips to enhance its combustion. The agro-materials were weighed, and blended together at the ratio of 40:30:30% for pig dung, palm kernel shell, and bamboo wood respectively for carbonization. Using a modified pyrolysis technique as described by Gunamantha and Widana (2018), and Okoli (2020), the weighed samples were fed into a clay pot of about 2.5-liters size, placed on the fire set-up, and covered with a lid to initiate anoxic combustion process. The materials were allowed to burn for about 6-8 hours at about 300-400 ^{o} C till no more smoke is being produced. The resultant product is then activated using the physical activation method by the application of water on the burning charcoal. The pot is removed from the fire, and the AC produced is allowed to cool. It is then spread on a clean slate to air dry before it is ground into powder. Thereafter, the activated charcoal is weighed again to ascertain its yield, and stored in a plastic container to reduce water absorption from the air.

Determination of Physic-chemical Properties and Mineral Concentration of AC

The physic-chemical characteristics such as the BD, SG, and WHC of the agro-waste derived AC was determined using the method described by Makinde and Sonaiya (2007), and modified by Omede *et al.* (2011). Bulk density is the weight of a material in a known volume of container. The specific gravity of a substance is a comparison of the density of the substance relative to a standard value (e.g., density of water). Specific gravity was determined as the ratio of the bulk density to that of water.

The pH of the AC was determined with the aid of a pH meter (HANNA Combo pH Meter, Model: HI 98129), while its mineral concentrations were determined after acid digestion using the Atomic Absorption Spectrophotometer (AAS) (Bulk Scientific, 205). Metals such as calcium (Ca), magnesium (Mg), potassium (K), phosphorus (P), sodium (Na), manganese (Mn), zinc (Zn), copper (Cu), iron (Fe), and chlorine (Cl) were measured using the AAS analysis.

Results and Discussion

The physical characteristics of the pig dung/bamboo stick/palm kernel shell derived AC product as summarized in Table 1 showed a slightly alkaline pH value at 8.49, with a high carbon content of 75.35% and total ash content of 13.13%. The pH value obtained was within the range of 6-10 reported for most agro-residue (wood and shells), and manure-derived activated charcoal particularly those produced at high temperatures as well as some commercially available AC products (Chen *et al.,* 2018; Boadu *et al.,* 2018). The pH variations among ACs occurs due to differences in their ash contents. Increased content of ash-rich minerals such as K, Ca, Na and Mg, and oxygen functional groups resulting from increased pyrolytic temperature have been observed to increase the pH values of ACs (Magdziarz *et al*., 2016; Ma *et al.,* 2017; Zhao *et al.,* 2017), giving it its slightly alkaline character. Reports suggest that lower pH activated charcoal products have more adsorption capacity compared to AC products with pH values of 12 (Hadi *et al.,* 2015; Bedia *et al.,* 2018). Therefore, the pH value obtained in this study was ideal for adsorption purposes as maximum adsorption of metals by most activated carbon occur at this pH (Nwabanne and Igbokwe, 2012).

The carbon content value recorded for the waste derived AC was 75.35%, while the total ash content value was 13.13%.

Table 1: Physical characteristics of Activated Charcoal

Parameter	Value
pH	8.49
Carbon content (%)	75.35
Total ash (%)	13.13
Bulk density (g/cm^3)	0.871
Water holding capacity	0.879
Specific gravity	0.725
Oil absorption capacity (g/g)	1.25
Percentage AC yield (%)	46.54

These values are similar to the submissions of Mozammel *et al.* (2002) who reported 76.32%, and 13.08% for fixed carbon, and ash content respectively. The increase in the carbon content of AC has been associated with the loss of the hydroxyl (–OH) surface as a result of dehydration during carbonization (Zielin´ska *et al.*, 2015). According to Domingues *et al.* (2017), higher carbon content ranging from 62.2 to 92.4% in ACs produced at higher carbonization temperatures were due to higher degrees of polymerization, which allows the formation of a more compact carbon structure in the resultant AC product (Lehmann and Joseph, 2009).

A good AC should have a high carbon, and low ash content produced from precursor materials with low inorganic content, moderately high volatility and density as found in the precursor materials used (Tadda *et al.,* 2016). Much lower carbon (26.31%), and ash content percentages (6.38%) have been recorded for PKS-based AC (Ma *et al.,* 2017) and bamboo-derived AC products (Yamauchi *et al.*, 2010), while Samanya and Yamauchi (2001) found AC-wood vinegar product to have ash content values of 13.40%.

Studies have also reported higher ash content values (22- 65%) for both manure, and wood-based ACs (Lima and Marshal, 2007; Yargicoglu *et al.,* 2014). Ash is considered an impurity, and therefore reduces the adsorptive capacity, and fixed carbon content of activated charcoal products at higher levels. According to Abdullah *et al.* (2000), AC products intended for adsorption should have ash levels not more than 20%.

The variations in carbon, and ash contents of an AC are influence by factors such as carbonization temperature, activation process/agent, and nature of material among others. Higher carbonization temperature will usually yield ACs with higher ash content, and decreased AC yield (Yorgun and Yildiz, 2015; Tadda *et al.,* 2016). Again, the structural composition (lignin, cellulose, hemicellulose etc.,) of the pyrolytic biomass as contained in pig dung, palm kernel shell, and bamboo, will also influence the physic-chemical properties such as ash, carbon content, and pH of the AC produced (Tomczyk *et al.,* 2020).

Results of the BD, WHC, and SG of the AC produced in this study were 0.871 g/cm^3, 0.879 g water/g AC and 0.725 respectively. The BD was slightly higher than the 0.64 g/cm^3, and 0.73 g/cm^3 reported by Evbuomwan *et al.* (2013), and Yargicoglu *et al.* (2014) for PKS-derived, and wood-derived ACs but similar with the 0.83 g/cm^3 reported by Hariprasad *et al.* (2016) for rice husk AC produced at high activation temperature (700°C). Bryne and Nagle (1997) reported a range of 0.06- 1.03 g/ cm^3 for wood-based ACs. High BD signifies increased mechanical strength or hardiness, which is a good attribute of activated charcoal required for water treatment/filtration processes (Zarifah, 2010). Adequate mechanical strength helps to reduce dust formation resulting from continuous friction between two AC particles (Vijayan *et al.,* 2012). Therefore, the hardiness of the AC obtained in this study is adequate, and lies within the recommended range for ideal sorbent materials.

The BD and WHC are the two bio-physical characteristics that could affect the nutritional value, and acceptability of animal feeds (Sundu *et al.*, 2008; Omede *et al.*, 2011) since they have correlation with feed intake, and storage volume in animals. Studies suggest that low BD diets significantly lowered the body weight of growing chicks as a result of decreased energy volume ratio of the diets (Shelton *et al.*, 2005), while diets with higher WHC values tend to absorb excess water within the gastro intestinal tract (GIT) of birds, which can trigger satiety, and low feed consumption in animals, and consequently causes poor animal performance (Kyriazakis and Emmans, 1995; Omede, 2010; Ohanaka *et al.*, 2017).

The BD, and WHC values of our waste- derived AC fell within the range reported for most conventional feedstuffs, and agro-waste derived ACs, and so may not negatively affect feed consumption when incorporated into poultry diets (Sundu *et al.,* 2008; Omede *et al.*, 2011).

Specific gravity of a powdered material is the ratio of the density of the material to the density of water. The SG value of 0.725 obtained in this study for AC was lower than SG values (1.26-1.61) reported for shell-derived activated charcoal, and ash (Fono Tamo *et al.*, 2014; Boadu *et al.,* 2018). Using specific gravity as a measure of physical quality of animal feeds, SG values of feed ingredients plays important roles in the transit of digesta particles through the gastro-intestinal tract of animals (Bhatti and Firkins, 1995; Ohanaka *et* *al.,* 2017). AC products with SG values less than 1 will traditionally float over water, and thus, will have lower retention time in the GIT of animals when ingested. However, SG value of AC obtained in this study, was much higher than the range of 0.33 – 0.46 reported for conventional feed resources and broiler rations produced in Nigeria (Omede, 2010) indicating it may impact diets after incorporation.

Activated charcoal yield is the ratio of the weight of dried activated charcoal to the weight of precursor materials carbonized. The AC yield of approximately 47% was higher when compared to those of shell-derived activated charcoals, and manure-based AC (Mozammel *et al.,* 2002; Lima and Marshal, 2007; Ma *et al.,* 2017). However, it is within the range reported for most activated charcoal derived from agro-waste. Increasing carbonization cum activation temperatures will result in the reduction of AC yield, while increasing its fixed carbon, and ash content due to increased volatilization during the carbonization process (Zarifah, 2010; Ahsan *et al.,* 2014).

Results also showed that AC produced from a blend of pig dung, PKS, and bamboo recorded oil adsorption capacity (OAC) value of 1.25 g/g meaning that 1 g of AC adsorbed 1.25 g of vegetable oil. The OAC of a given sorbent is dependent on the surface chemistry, porosity, bulk density, its affinity for oil and the nature/type of oil sorbate to be adsorbed (Bandura *et al.,* 2017). Oil adsorption capacity decreases with increased bulk density of a sorbent while low BD favors the formation of capillaries which efficiently absorbs oil within its pores (Angelova *et al.,* 2011). However, the OAC value obtained in this study compares favourably with the range reported for most commercial sorbents, and will efficiently adsorb oil from soil/water surface (Zadaka-Amir *et al.,* 2013; Bandura *et al.,* 2015).

Mineral composition of activated charcoal

The mineral characteristics of AC is summarized in Table 2. The study showed that the major metals in AC were phosphorus (25,100.66 mg/kg), potassium (9,300.29 mg/kg), calcium (5,550.28 mg/kg), magnesium (4410.10 mg/kg), sodium (1405.18 mg/kg), and iron (1304.65 mg/kg) with chlorine recording the least concentration at 25.46 mg/kg. Several studies have found some level of mineral concentration in ACs produced from different agro-waste, and residues. The concentration of minerals obtained in this study were much higher than the values reported for PKS-derived activated charcoal (Evbuomwan *et al.,* 2013) but lower than the values reported for wood, and coal-base activated charcoal (Machida *et al.,* 2005).

Table 2: Mineral composition of activate charcoal

Parameter	Result
Calcium (mg/kg)	5550.28
Phosphorus (mg/kg)	25100.66
Sodium (mg/kg)	1405.18
Magnesium (mg/kg)	4410.10
Potassium (mg/kg)	9300.29
Manganese (mg/kg)	671.33
Iron (mg/kg)	1304.65
Zinc (mg/kg)	103.48
Copper (mg/kg)	39.46
Chlorine (mg/kg)	25.46

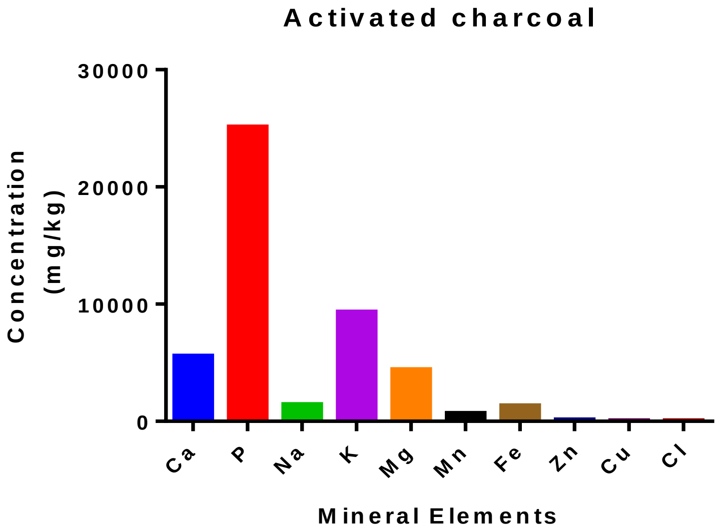

Figure 1: Elemental Concentration of Agro-waste Derived Activated Charcoal

The variations in the mineral contents of AC may be linked to the biomass composition, and carbonization temperature, which destroys the carbon structure, and influences the level of ash produced.

The levels of P, and K (figure I) were much higher in the waste-derived AC compared to other mineral fractions. This may have been due to the higher ratio of pig dung to the other feedstocks (PKS, and bamboo), as similar reports of increased P, and K contents were observed in AC produced from pig dung feedstock (Lima and Marshal, 2007). Reports have also suggested that the higher concentration of phosphorus entrapped within the carbon matrix of manure-based activated charcoal as polyphosphate anions increases the adsorption capacity of such AC for heavy metals (Xu *et al.*, 2013; Lima *et al.*, 2015). The K concentration in the AC produced surpassed the minimum dietary requirement for poultry production. It is therefore expected that the incorporation of the AC into poultry feeds will result in increase in P, and K content of the feeds.

The K mineral, is often called the alkalizer and necessary for the maintenance of growth performance, nutrient utilization, bone and muscle development in broilers (Mushtaq *et al.*, 2013; Ohanaka *et al.*, 2017). However, at higher levels, K can cause disturbances in the acid-base balance of broiler chicken through dietary electrolyte balance (dEB) disruptions (Ohanaka, 2016). The Mg, and Ca concentrations of AC obtained in this study showed that AC can supply the Mg, and half the Ca requirements for broiler production (Aviagen, 2019), which reduces the cost of supplying the mineral needs of the birds from other sources. The Na concentration in the AC (1400 mg/kg) could adequately support early broiler chick's development (Jankowski *et al.*, 2011). Na plays a significant role in stimulating feed intake, and digestive enzymes functions (Mushtaq *et al.*, 2013).

The most abundant micro minerals in the AC produced is iron, Manganese and zinc with levels more than required to support poultry production (SON, 2018; Aviagen, 2019). The level of minerals contained in the AC is influenced by several factors which include (1) the nature, and condition of feedstock during pyrolysis, (2) the temperature during carbonization, and activation, and (3) the AC production method (Tadda *et al.*, 2016). Generally, the inclusion of this activated charcoal product as an additive in poultry diets could be an alternative mineral source or at best compliment the use of the more expensive conventional mineral sources thereby lowering the cost of poultry production for small holder farmers in tropics. However, there is a need to correct for mineral ratio imbalances that may arise when incorporated in animal feeds owing to its high content of P, and K. However, its inclusion in feeds could improve the performance of livestock through GIT microbiome modifications (Willson *et al.*, 2019), as a result of its physic-chemical and adsorptive properties.

Conclusion

This study showed that carbonizing a blend of agro-waste such as pig dung, PKS, and bamboo wood could yield activated charcoal product with quality characteristics, and content of minerals that can ably support sustainable crop, and livestock production in the tropics. The AC had satisfactory physic-chemical properties within the range of most AC products that can adsorb contaminants from animal feeds, GIT, soil/water surfaces, and can also be applied in other industrial operations.

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